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Indian Standard

SPECIFICATION FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

(*First Revision*)

UDC 661·833·452 : 621·793·3

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6

May 1988

Indian Standard

SPECIFICATION FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

(First Revision)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 12 May 1987, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally published in 1979. Subsequently, it was decided by the Electroplating Chemicals Sectional Committee to revise this standard to bring in line with international practices and to incorporate the experience gained by manufacturers and users of this material during these years.

0.3 Hypophosphites are powerful reducing agents, precipitating metals from metal salt solutions. Sodium hypophosphite is used as reducing agent for nickel cobalt and palladium chlorides in electroless nickel, cobalt and palladium plating applications. In this revision, a new grade of sodium hypophosphite has been introduced which is required for special applications of electroless plating for electronic industries.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for sodium hypophosphite required for electroless plating applications.

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*Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 Sodium hypophosphite shall be of the following two grades:

a) Grade 1 — Used for commercial applications of electroless plating; and

b) Grade 2 — Used for special applications of electroless plating for electronic industries.

3. REQUIREMENTS

3.1 Description — Sodium hypophosphite of both grades shall be in the form of white crystalline solid, readily soluble in water, free from foreign matter and other visible impurities, and shall correspond essentially to the formula $NaH_2PO_2.H_2O$.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is indicated in column 5 of Table 1.

4. HANDLING

4.1 The presence of dust or any other foreign matter in sodium hypophosphite may cause decomposition of the bath and hence, the material shall be kept in well closed air-tight containers. While opening, adequate care shall be taken to prevent foreign particles from falling into the material and the lid shall be replaced soon after withdrawing the material.

4.2 Since sodium hypophosphite is a very strong reducing agent, the following precautions shall be observed while using the material:

a) Avoid prolonged or repeated skin contact,

b) Avoid direct contact since it irritates eyes, and

c) In cases of skin or eyes coming in contact with this material, wash thoroughly with plenty of water.

5. PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be supplied in air-tight containers of the capacity agreed to between the purchaser and the manufacturer. The containers shall be closed preferably with replaceable closure.

TABLE 1 REQUIREMENTS FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

(*Clause* 3.2)

SL No.	CHARACTERISTIC	REQUIREMENT OF		METHOD OF TEST (REF TO CLAUSE No. IN APPENDIX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Matter insoluble in water, percent by mass, *Max*	0·01	0·01	A-2
ii)	Chloride (as Cl), percent by mass, *Max*	0·02	0·012	A-3
iii)	Sulphate (as SO_4), percent by mass, *Max*	0·02	0·009	A-4
iv)	Arsenic (as As), percent by mass, *Max*	0·005	0·000 12	A-5
v)	Calcium (as Ca), percent by mass, *Max*	0·005	0·002 5	A-6
vi)	Heavy metals (as Pb), percent by mass, *Max*	0·005	—	A-7
vii)	Iron (as Fe), percent by mass, *Max*	0·005	0·000 2	A-8
viii)	Lead + Copper + Zinc (as Pb + Cu + Zn), percent by mass, *Max*	—	0·000 05	A-9
ix)	Molybdenum + Cadmium + Mercury (as Mo + Cd + Hg), percent by mass, *Max*	—	0·000 005	A-9
x)	Sodium hypophosphite (as $NaH_2PO_2.H_2O$), percent by mass, *Min*	98·0	99·5	A-10

5.2 Marking

5.2.1 The containers shall be marked with the following particulars:

a) Manufacturer's name and/or his recognized trade-mark, if any;

b) Net mass of the material;

c) Name and grade of the material;

d) Month and year of manufacture; and

e) Batch number.

5.2.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING

6.1 The method of preparing representative samples of the material and criteria for its conformity with this specification, shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2 and *Table* 1)

METHOD OF TEST FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall, mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-2.1 Principle

A-2.1.1 This method is based on dissolving a known quantity of material in water followed by filtering and weighing any insoluble matter left behind.

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*Specification for water for general laboratory use (*second revision*).

A-2.2 Procedure

A-2.2.1 Weigh accurately 10·0 g of the material, transfer to a 250 ml beaker and dissolve it in 100 ml of water. Heat on a steam-bath for 1 hour with occasional stirring. Filter quantatively through a sintered glass crucible (G No. 4), wash well with hot water and dry at 105°C ± 2°C. Cool in a desiccator and weigh.

A-2.3 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the residue.

A-3. DETERMINATION OF CHLORIDE

A-3.1 Principle

A-3.1.1 This method is based on dissolving a known quantity of material in water and the chloride present in the solution is precipitated as silver chloride followed by filtering and weighing the precipitate.

A-3.2 Reagents

A-3.2.1 *Silver Nitrate Solution* — 1·7 percent (*m/v*) in water.

A-3.2.2 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-3.3 Procedure

A-3.3.1 *Grade 1 — Sodium Hypophosphite*

A-3.3.1.1 Weigh accurately about 5·0 g of the material and dissolve in 20 ml of water. Add 5 ml of silver nitrate solution and 10 ml of concentrated nitric acid. Heat gently to expel oxides of nitrogen. Allow the contents to stand for 2 hours, filter the silver chloride precipitate through a sintered glass crucible (G No. 4), wash well with hot water and dry the crucible at 120°C ± 2°C. Cool the crucible in a desiccator and weigh.

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*Specification for nitric acid (*second revision*).

A-3.3.1.2 *Calculation*

$$\text{Chloride (as Cl), percent by mass} = \frac{24.74}{M_1} \times M_2$$

where

M_1 = mass in g of the material taken, and

M_2 = mass in g of silver chloride precipitate.

A-3.3.2 *Grade 2 — Sodium Hypophosphite*

A-3.3.2.1 Weigh accurately about 10·0 g of the material and dissolve in 40 ml of water. Add 10 ml of silver nitrate solution and 20 ml of concentrated nitric acid, and proceed by method as described in **A-3.3.1.1** for determining chloride content.

A-3.3.2.2 *Calculation*

$$\text{Chloride (as Cl), percent by mass} = \frac{24.74}{M_1} \times M_2$$

where

M_1 = mass in g of the material taken, and

M_2 = mass in g of silver chloride precipitate.

A-4. DETERMINATION OF SULPHATE

A-4.1 Principle

A-4.1.1 This method is based on dissolving a known quantity of material in water and the sulphate present in the solution is precipitated as barium sulphate followed by filtering and weighing the precipitate.

A.4.2 Reagents

A-4.2.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-4.2.2 *Barium Chloride Solution* — 12 percent (*m/v*) in water.

A-4.3 Procedure

A-4.3.1 *Grade 1 — Sodium Hypophosphite*

A-4.3.1.1 Weigh accurately 10·0 g of the material, transfer to 250 ml beaker and dissolve it in 100 ml of water. Heat on a steam

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*Specification for hydrochloric acid (*second revision*).

bath for 1 hour with occasional stirring. Filter through a sintered glass crucible (G No. 4), wash well with water to remove any insoluble matter left behind.

A-4.3.1.2 To the filtrate obtained in **A-4.3.1.1**, add 1 ml of concentrated hydrochloric acid and heat to boiling. Add 5 ml of barium chloride solution with stirring and allow to stand overnight. Filter the precipitate through a sintered glass crucible (G No. 4), wash well with hot water and dry at 105°C ± 2°C. Cool in a desiccator and weigh.

A-4.3.1.3 *Calculation*

$$\text{Sulphate (as } SO_4\text{), percent by mass} = \frac{41{\cdot}15}{M_1} \times M_2$$

where

M_1 = mass in g of the material taken, and
M_2 = mass in g of the barium sulpate precipitate.

A-4.3.2 *Grade 2 — Sodium Hypophosphite*

A-4.3.2.1 Weigh accurately 20·0 g of the material, transfer to 500 ml beaker and dissolve it in 200 ml of water. Heat on a steam bath for 1 hour with occasional stirring. Filter through a sintered glass crucible (G No. 4), wash well with hot water to remove any insoluble matter left behind. Using the filtrate, proceed by method as described in **A-4.3.1.2** for determining sulphate content.

A-4.3.2.2 *Calculation*

$$\text{Sulphate (as } SO_4\text{) percent by mass} = \frac{41{\cdot}15}{M_1} \times M_2$$

where

M_1 = mass in g of the material taken, and
M_2 = mass in g of the barium sulphate precipitate.

A-5. DETERMINATION OF ARSENIC

A-5.1 Grade 1 — Sodium Hypophosphite

A-5.2 Reagents

A-5.2.1 *Concentrated Nitric Acid — See* IS : 264-1976*.

A-5.2.2 *Concentrated Sulphuric Acid — See* IS : 266-1977†.

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*Specification for nitric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

A-5.3 Procedure

A-5.3.1 Weigh accurately 1·0 g of the material, transfer to an evaporating dish and dissolve in 10 ml of water. Add 5 ml of concentrated nitric acid and evaporate to dryness on a steam bath. Re-evaporate with 10 ml of water and 5 ml of concentrated sulphuric acid, and continue the process till the strong acid fumes are completely removed. Cautiously add 10 ml of water and proceed by the silver diethyl dithiocarbomate method to determine arsenic content as prescribed in IS : 2088-1983*.

A-5.3.2 *Grade 2 — Sodium Hypophosphite*

A-5.3.2.1 For the determination of arsenic, follow the method as given in IS : 11124-1984†.

A-6. DETERMINATION OF CALCIUM

A-6.1 Principle

A-6.1.1 This method is based on dissolving a known quantity of material in water followed by treatment with ammonium hydroxide and ammonium oxalate solutions, and the turbidity produced by the solution is compared with turbidity produced by the control standard test solution to determine calcium content.

A-6.2 Reagents

A-6.2.1 *Ammonium Hydroxide* — Relative density 0·9 approximately.

A-6.2.2 *Ammonium Oxalate Solution* — 4 percent (*m/v*) in water.

A-6.2.3 *Standard Calcium Solution* — Weigh accurately 0·250 g of calcium carbonate, previously dried at 200°C. Dissolve in 10 ml of water using 1 ml of concentrated hydrochloric acid. Heat to boiling, cool and dilute with water to the mark in a 1 000 ml volumetric flask. One millilitre of this solution is equivalent to 0·1 mg of calcium (as Ca).

A-6.3 Procedure

A-6.3.1 *Grade 1 — Sodium Hypophosphite*

A-6.3.1.1 Weigh accurately 2·0 g of the material and dissolve in 20 ml of water. Warm at 50°C, add 1 ml of ammonium hydroxide and

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*Methods for determination of arsenic (*second revision*).

†Method for atomic absorption spectrophotometric determination of arsenic.

2 ml of ammonium oxalate solution. Simultaneously carry out a control test by adding to 15 ml of warm (50°C) water, 1 ml of standard calcium solution, 1 ml of ammonium hydroxide followed by 2 ml of ammonium oxalate solution. Allow the solutions to stand for 10 minutes.

A-6.3.1.2 The material shall be taken as having satisfied the requirement of the test if the turbidity produced by the material is not greater than that produced in the control test solution.

A-6.3.2 *Grade 2 — Sodium Hypophosphite*

A-6.3.2.1 Weigh accurately 2·0 g of the material and dissolve in 20 ml of water. Warm at 50°C, add 1 ml of ammonium hydroxide and 2 ml of ammonium oxalate solution. Simultaneously carry out a control test by adding to 10 ml of warm (50°C) water, 0·5 ml standard calcium solution, 0·5 ml of ammonium hydroxide followed by 1 ml ammonium oxalate solution. Allow solutions to stand for 10 minutes.

A-6.3.2.2 The material shall be taken as having satisfied the requirement of the test if the turbidity produced by the material is not greater than that produced in the control test solution.

A-7. DETERMINATION OF HEAVY METALS

A-7.1 Principle

A-7.1.1 This method is based on dissolving a known quantity of material in water followed by separation of solution into 2 parts (*see* **7.4.1**) and treat both solutions with lead standard solution, hydrochloric acid and hydrogen sulphide solution. The colours produced by the two solutions are compared with each other to determine lead content.

A-7.2 Apparatus

A-7.2.1 *Nessler Cylinder* — Two, each of 50 ml capacity (*see* IS : 4161-1967*).

A-7.3 Reagents

A-7.3.1 *Dilute Hydrochloric Acid* — Approximately 1 N.

A-7.3.2 *Hydrogen Sulphide Solution* — A freshly prepared, saturated aqueous solution of hydrogen sulphide gas.

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*Specification for Nessler cylinders.

A-7.3.3 *Standard Lead Solution* — Dissolve 0·16 g of lead nitrate in 100 ml of water containing 1 ml of concentrated nitric acid and dilute with water up to the mark in a 100 ml volumetric flask. Dilute 1 ml of this solution to the mark in a 100 ml volumetric flask. One millilitre of this dilute solution is equivalent to 0·01 mg of lead (as Pb).

A-7.4 Procedure

A-7.4.1 Weigh accurately 2·0 g of the material, dissolve in 30 ml of water and dilute up to the mark with water in a 50 ml volumetric flask. Transfer 30 ml of this solution to a Nessler cylinder A and 10 ml to Nessler cylinder B. Add 4 ml of the standard lead solution to Nessler cylinder B. Then add 1 ml of dilute hydrochloric acid followed by 10 ml of hydrogen sulphide solution to each of the cylinders and dilute up to the mark with water.

A-7.4.2 The material shall be taken as having satisfied the requirement of the test if the solution in Nessler cylinder A is not darker than that in Nessler cylinder B.

A-8. DETERMINATION OF IRON

A-8.1 Principle

A-8.1.1 This method is based on dissolving a known quantity of material in water followed by evaporation with nitric acid and the resultant residue is dissolved in water, sulphuric acid, ammonium persulphate and butanolic potassium thiocyanate solutions. Red colour produced in the butanolic layer of the solution is compared with the red colour produced by the control standard test solution to determine iron content.

A-8.2 Apparatus

A-8.2.1 *Nessler Cylinders* — Two of 50 ml capacity (*see* IS : 4161-1967*).

A-8.3 Reagents

A-8.3.1 *Concentrated Nitric Acid* — *See* IS : 264-1976†.

A-8.3.2 *Concentrated Sulphuric Acid* — *See* IS : 266-1977‡.

A-8.3.3 *Ammonium Persulphate*

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*Specification for Nessler cylinders.

†Specification for nitric acid (*second revision*).

‡Specification for sulphuric acid (*second revision*).

A-8.3.4 *Butanolic Potassium Thiocyanate* — Dissolve 10 g of pottassium thiocyanate in 10 ml of water. Warm the solution to 25 to 30°C, and add *n*-butanol to measure 100 ml and shake vigorously.

A-8.3.5 *Standard Iron Solution I* — Dissolve 0·702 g of ferrous ammonium sulphate $(NH_4)_2SO_4.FeSO_4.6H_2O$ in water containing 10 ml of concentrated sulphuric acid and dilute with water to 100 ml in a 1 000 ml volumetric flask. Dilute 10 ml of this solution with water to the mark in a 100 ml volumetric flask just before use. One millilitre of this diluted solution is equivalent to 0·01 mg of iron (as Fe).

A-8.3.6 *Standard Iron Solution II* — Dissolve 1·404 g of ferrous ammonium sulphate $(NH_4)_2SO_4.FeSO_4.6H_2O$ in water containing 20 ml of concentrated sulphuric acid and dilute with water to the mark in a 1 000 ml volumetric flask. Dilute 1 ml of this solution to the mark 1 000 ml in volumetric flask just before use. One millilitre of this diluted solution is equivalent to 0·000 2 mg of iron (as Fe).

A-8.4 Procedure

A-8.4.1 *Grade 1 — Sodium Hypophosphite*

A-8.4.1.1 Weigh accurately 1·0 g of the material and dissolve in 10 ml of water. Add 5 ml of concentrated nitric acid and evaporate on steam bath to dryness. Transfer the residue with 15 ml of water and 2 ml of concentrated sulphuric acid to a 50 ml Nessler cylinder. Add about 50 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Shake well and allow the layers to separate. Carryout a control test in another Nessler cylinder using 5 ml of standard iron solution I and the same quantities of reagents in the same total volume as with the material.

A-8.4.1.2 The material shall be taken as having satisfied the requirement of the test if the intensity of red colour produced in butanol layer is not greater than in the control test solution.

A-8.4.2 *Grade 2 — Sodium Hypophosphite*

A-8.4.2.1 Weigh accurately 1·0 g of the material and dissolve in 10 ml of water. Add 5 ml of concentrated nitric acid and evaporate on steam bath to dryness. Transfer the residue with 15 ml of water and 2 ml of concentrated sulphuric acid to a 50 ml Nessler cylinder. Add about 50 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Shake well and allow the layers to separate. Carry out a control test in another Nessler cylinder using 10 ml of standard iron solution II and the same quantities of reagents in the same total volume as with the material.

A-8.4.2.2 The material shall be taken as having satisfied the requirement of the test if the intensity of red colour produced in butanol layer is not greater than in the control test solution.

A-9. DETERMINATION OF LEAD, COPPER, ZINC, MOLYBDENUM, CADMIUM AND MERCURY BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

A-9.1 Outline of the Method

A-9.1.1 The metallic elements in the ground state will absorb light of the same wavelength (*see* Table 2) as they emit, when excited . When a radiation from a given excited element is passed through a flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state element in the flame. A hollow cathode lamp, whose cathode is made up of the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant-fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photo sensitive device measures the attenuated transmitted radiation.

TABLE 2 OPERATING CONDITIONS FOR ATOMIC ABSORPTION SPECTROPHOTOMETRY

(*Clauses* A-9.1.1 *and* A-9.8.4)

SL No.	METAL	WAVE LENGTH (nm)	FUEL	OXIDANT
i)	Lead	283·3	Acetylene	Air
ii)	Copper	324·7	Acetylene	Air
iii)	Zinc	213·8	Acetylene	Air
iv)	Molybdenum	313·3	Acetylene	Air
v)	Cadmium	228·8	Acetylene	Air
vi)	Mercury	253·6	Acetylene	Air

A-9.2 Apparatus

A-9.2.1 *Atomic Absorption Spectrophotometer* — The instrument shall consist of an atomizer and a burner with suitable pressure—regulating devices capable of maintaining a constant oxidant-fuel pressure for the test. It shall also consists of:

a) a hollow cathode lamp for each metal to be tested,

b) an optical system capable of isolating the desired line of radiation,

c) an adjustable slit,

d) a photomultiplier tube or any other photo-sensitive device as a light measuring and amplifying device, and

e) a read-out mechanism for indicating the amount of absorbed radiation.

NOTE — Multi-element hollow cathode lamps are available and found satisfactory.

A-9.3 Oxidant

A-9.3.1 Air is the usual oxidant used and it shall be cleaned and dried through a suitable filter to remove oil, water and other foreign substances.

A-9.4 Fuel (Acetylene)

A-9.4.1 Commercially available acetylene is the usual fuel used.

A-9.5 Reagents

A-9.5.1 *Concentrated Hydrochloric Acid* — Sp.gr 1·19 approximately (*see* IS : 265-1976*).

A-9.5.2 *Concentrated Nitric Acid* — Sp.gr 1·42 approximately (*see* IS : 264-1976†).

A-9.6 Standard Solutions

A-9.6.1 *Lead* (1 ml = 0·5 mg of Pb) — Dissolve 500 mg of pure lead in a minimum quantity of nitric acid (1 : 1 by volume). Transfer the solution to a 1-litre volumetric flask and dilute up to the mark with water.

A-9.6.2 *Copper* (1 ml = 0·1 mg of Cu) — Dissolve 100 mg of pure electrolytic copper in a minimum quantity of nitric acid (1 : 1 by volume). Transfer the solution to a 1-litre volumetric flask and dilute up to the mark with water.

A-9.6.3 *Zinc* (1 ml = 0·1 mg of Zn) — Dissolve 100 mg of pure zinc in a minimum quantity of nitric acid (1 : 1 by volume). Transfer the

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*Specification for hydrochloric acid (*second revision*).

†Specification for nitric acid (*second revision*).

solution to a 1-litre volumetric flask and dilute up to the mark with water.

A-9.6.4 *Molybdenum* (1 ml = 0·1 mg of Mo) — Dissolve 100 mg of pure molybdenum in a minimum quantity of nitric acid (1 : 1 by volume). Transfer the solution to a 1-litre volumetric flask and dilute up to the mark with water.

A-9.6.5 *Cadmium* (1 ml = 0·1 mg of Cd) – Dissolve 0·203 1 g of A.R. cadmium chloride ($CdCl_2.2H_2O$) in 200 ml of water. Transfer the solution to a 1-litre volumetric flask and dilute with water.

A-9.6.6 *Mercury* (1 ml = 0·1 mg of Hg) — Dissolve 100 mg of pure mercury in a minimum quantity of nitric acid (1 : 1 by volume). Transfer the solution to a 1-litre volumetric flask and dilute up to the mark with water.

A-9.7 Calibration

A-9.7.1 Prepare at least four standard solutions to bracket the expected metal concentration in the sample, by diluting the standard metal solutions with water.

A-9.7.2 Optimize the response of the instrument by adjusting the burner height and flame. Apply the current to the cathode lamp as suggested by the manufacturer. Allow the instrument to warm up until the energy source stablizes. The time required for this process may vary from 10 to 20 minutes.

A-9.7.3 Set the slit width for the metal of interest based on the manufacturer's instructions. Feed the fuel and the oxidant for the metal of interest as indicated in Table 2 to the burner and ignite the emitted mixture. Adjust the fuel and the oxidant pressure in accordance with the procedure given by the manufacturer to give maximum sensitivity for a given concentration of the metal.

A-9.7.4 Atomize the water and check the aspiration rate for a period of 2 minutes. Normally, an aspiration rate of 4 to 5 ml per minute is satisfactory. The standard and samples of the metal of interest should have the same aspiration rate.

A-9.7.5 Set the wavelength to the approximate value as given in Table 2 depending on the metal of interest. Atomize the standard solution and select the most sensitive wavelength setting for maximum absorption. The wavelength indicated in Table 2 are the most sensitive recommended values for the respective metals. Once the atomization is completed, record the absorbance reading. This procedure has to be repeated for each standard solution.

A-9.7.6 Prepare a calibration curve relating the absorbance and the amount of metal in microgrammes present in the standard solutions.

A-9.8 Procedure

A-9.8.1 Proceed in accordance with **A-9.7.1** to **A-9.7.6** for preparing the calibration curve. This procedure has to be repeated for each metal.

A-9.8.2 Rinse the atomizer by aspirating water.

A-9.8.3 Weigh accurately 1 g of the material, transfer to 250 ml beaker and dissolve in 50 ml of water. Heat on a steam bath for 1 hour with occasional stirring. Filter through a sintered glass crucible (G No. 4) and wash well with water to remove any insoluble matter left behind. Retain the filtrate for determining metallic impurities.

A-9.8.4 Take the filtrate from **A-9.8.3** and atomize the metal of interest from the above solution at a wavelength and operating conditions listed in Table 2 and determine its absorbance. Mass of metal in microgrammes may be determined from the calibration curve.

A-9.9 Calculation

$$\text{Metal, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material taken, and

M_2 = mass in g of the metal found from the calibration curve.

A-10. DETERMINATION OF SODIUM HYPOPHOSPHITE

A-10.1 Principle

A-10.1.1 This method is based on dissolving a known quantity of material in water followed by oxidation with bromine and then treated with sulphuric acid and potassium iodide solutions. The liberated iodine (represents the excess of bromine added) is titrated against sodium thiosulphate to determine sodium hypophosphite content.

A-10.2 Reagents

A-10.2.1 *Standard Bromine Solution* (Approximately 0·1 N) — Dissolve 10·7 g of potassium bromate and 2·5 g of potassium bromide in 40 ml of water. Transfer to a 100 ml volumetric flask and dilute with water up to the mark. Measure accurately from a burette 30 to 35 ml of the solution into a glass stoppered conical flask and dilute with 120 ml of water and proceed with standardization as indicated in **A-10.3** to determine its normality.

A-10.2.2 *Dilute Sulphuric Acid* — 10 percent (*v/v*).

A-10.2.3 *Potassium Iodide Solution* — 20 percent (*m/v*).

A-10.2.4 *Standard Sodium Thiosulphate Solution* — Approximately 0·1 N.

A-10.2.5 *Starch Indicator Solution* — Mix 5 g of starch and 0·01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid.

A-10.3 Procedure

A-10.3.1 Weigh accurately 0·3 g of the material, dissolve in water and dilute up to the mark with water in a 200 ml volumetric flask. Transfer 50 ml of this solution to a 500 ml glass stoppered conical flask and add 50 ml of standard bromine solution and 120 ml of water. Then add 20 ml of dilute sulphuric acid and stopper immediately. Shake gently and allow to stand for 3 hours. Add quickly 10 ml of potassium iodide solution and titrate the liberated iodine (representing the excess of bromine) with standard sodium thiosulphate solution until the solution is pale yellow. Then add starch indicator solution and continue the titration till the blue colour is just discharged.

A-10.4 Calculation

$$\text{Sodium hypophosphite (as } NaH_2PO_2.H_2O \text{), percent by mass} = \frac{2{\cdot}651\,(\,50\,N - VN_1\,)}{M}$$

where

N = Normality of standard bromine solution,

V = volume in ml of standard sodium thiosulphate solution consumed in titration,

N_1 = normality of standard sodium thiosulphate solution, and

M = mass in g of material in the aliquot taken for the test.

APPENDIX B

(*Clause* 6.1)

SAMPLING OF SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing and handling test samples, the following precaution shall be observed.

B-1.1 The samples shall not be taken at a place exposed to weather.

B-1.2 The samples shall be placed in suitable, dry and air-tight containers preferably with replaceable closure.

B-1.3 While opening a sample container, adequate care shall be taken to prevent foreign particles from falling into the material.

B-1.4 Each samples container shall be marked with full details of sampling, the date of sampling, grade, the year of manufacture and the batch number, if any.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested from each lot separately.

B-2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 3.

TABLE 3 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE, N	SAMPLE SIZE, n
(1)	(2)
Up to 50	3
51 to 100	4
101 to 300	5
301 to 500	7
501 and above	10

B-2.3.1 These containers shall be selected at random from the lot. In order to ensure randomness of selection, reference may be made to IS : 4905-1968*. In case this standard is not readily available, the following procedure may be adopted:

> Starting from any container in the lot, count them in one order as 1, 2, 3,...........up to r and so on where r is the integral part of N/n (N and n being the lot size and the sample size respectively). Every rth container thus counted shall be withdrawn till the requisite number of containers is obtained.

B-3. TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a small quantity of material shall be taken from different parts of the container. The quantity of material so drawn from each container shall be sufficient to make triplicate determinations for all the requirements given in Table 1 of the specification.

B-3.2 Out of these portions, approximately equal quantities of material shall be taken and mixed thoroughly so as to form a composite sample of quantity sufficient to make triplicate determinations for all the characteristics to be tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the manufacturer and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each selected container shall be divided into three equal parts each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual samples and the composite samples shall be transferred to separate sample containers. All the containers shall be sealed air-tight and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite and a set of individual samples shall bear the seals of both the purchaser and the manufacturer. They shall be kept at a place as agreed to between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for determining sodium hypophosphite content shall be conducted on each of the individual samples.

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*Methods of random sampling.

B-4.2 Tests for the remaining requirements, prescribed in Table 1, shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 Test on Individual Samples — From the test results for sodium hypophosphite content, the mean (X) and the range (R) of the test results shall be computed (R being defined as the difference between the maximum and minimum values of the test results). The lot shall be declared to have satisfied this requirement, if the expression ($X—0{\cdot}6\,R$) is greater than or equal to 98·0.

B-5.2 Tests on the Composite Sample — The lot shall be considered to have passed in respect of the characteristics tested on the composite sample (*see* **B-4.2**), if all the test results on the composite sample satisfy requirements given in Table 1.

B-5.3 The lot shall be declared as conforming to the requirements of this specification, if **B-5.1** and **B-5.2** are satisfied.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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IS 9342 : 1987 SPECIFICATION FOR SODIUM HYPOPHOSPHITE FOR ELECTROLESS PLATING

(First Revision)

(*Page* 18, *clause* **A-10.2.1**) — Substitute the following for the existing:

'**A-10.2.1** *Standard Bromine Solution* (Approximately 0.1 N) – Dissolve 2.784 g of anhydrous potassium bromate ($KBrO_3$) in water. Add 10 g of potassium bromide (KBr) crystals, dissolve and dilute to 1 000 ml. This will give 0.1 N bromate bromine solution.'

(CHD 1)

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